PATENT

Atty. Docket No. NNM-001

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANTS:

Mayes and Tyler

SERIAL NUMBER:

09/308,166

GROUP NUMBER:

2754

FILING DATE:

June 25, 1999

EXAMINER:

Not Yet Assigned

TITLE:

Magnetizable Device

CERTIFICATE OF FACSIMILE AND EXPRESS MAILING

I hereby certify that this correspondence and any document(s) referred to as attached hereto is being transmitted to Ms. Patricia Booker at the U. S. Patent and Trademark Office via facsimile (1-703-746-6695) and deposited with the United States Postal Service as Express Mail (Label No. **EL956539515US**), addressed to: Box PCT, Commissioner of Patents, Washington, DC 20231, ATTN: Ms. Patricia Booker, on June 19, 2002.

Date

-/-

Brenda T. Kowalczuk

Box PCT

Commissioner for Patents Washington, D.C. 20231 Attn: Ms. Patricia Booker

Sir:

Submitted herewith is/are:

Transmittal Form (1 pg.); Letter to Office of PCT Operations (3 pgs.); Exhibit A (1 pg.); Exhibit B (2 pgs.); Exhibit C (1 pg.); Exhibit D (1 pg.); Exhibit E (4 pgs.); Exhibit F (10 pgs.); Associate Power of Attorney (1 pg.); and a postcard.

	1		Application	Serial Number	09/30	08,166		
TRANSMITTAL			Filing Date		June	June 25, 1999		
			First Named Inventor		Mayo	Mayes		
			Group Art Unit		2754	2754		
			Examiner Na	ame	Not y	vet assigned		
•	FORM		Attorney Do	cket No.	NNN	1-001		
			Patent No.		Not a	applicable		
			Issue Date	Not applicable		pplicable		
		ENC	LOSURES (ci	heck all that apply)				
☐ Fee	Transmittal Form			ce to File Missing		Notice of Appeal to Board		
		_		ication (PTO-1553)		of Patent Appeals and Interferences		
	☐ Check Attached☐ Copy of Fee Transmittal Form		Formal Draw	ings		Appeal Brief (in triplicate)		
	Amendment and Response		Request For C Examination Transmittal			Status Inquiry		
	☐ Preliminary ☐ After Final	_			\boxtimes	Return Receipt Postcard		
	Affidavits/declaration(s) Letter to Official Draftsperson	Associate Pov		wer of Attorney		Certificate of First Class Mailing under 37 C.F.R. 1.8		
including Drawings [Total Sheets]			Terminal Disclaimer			Certificate of Facsimile and Express Mailing		
	Petition for Extension of Time	of Attorney f		claration and Power or Utility or Design		Additional Enclosure(s) (please identify below)		
		• •			er to Office of PCT Operations			
☐ Information Disclosure ☐ Small Entity Statement		ity Statement 2		ibits A-F				
[Form PTO-1449 Copies of IDS Citations	computer	CD(s) for large table or program					
	Certified Copy of Priority		Amendment A	After Allowance				
	Document(s) Sequence Listing submission Paper Copy/CD Computer Readable Copy Statement verifying identity of above		Request for C Correction Certificat duplicate)	Certificate of e of Correction (in				
CORRESPONDENCE ADDRESS			SIGNATURE BLO	OCK	D (CH)			
Direct all correspondence to: Patent Administrator Testa, Hurwitz & Thibe High Street Tower 125 High Street Boston, MA 02110 Tel. No.: (617) 248-710 Fax No.: (617) 248-710		000	High Street Tower 125 High Street		Mark L. Beloborodov Attorney for Applicants Testa, Hurwitz & Thibeault, LLP High Street Tower			

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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EXAMINER:

Not Yet Assigned

TITLE:

Magnetizable Device

Box PCT Commissioner of Patents Washington, DC 20231 Attn: Ms. Patricia Booker

LETTER TO OFFICE OF PCT OPERATIONS

- 1. On November 17, 1997, Applicants filed an international patent application claiming priority to Great Britain Patent Application Serial No. 9623851.4, which was filed on November 16, 1996. The international application was assigned International Application No. **PCT/GB97/03152**.
- 2. On May 28, 1998, International Application No. **PCT/GB97/03152** was published under PCT Article 21(2) in the English language as International Publication No. WO98/22942. A copy of the title page of the published international application is attached hereto as Exhibit A.
- 3. On May 14, 1999, Applicants requested entry for International Application No. PCT/GB97/03152 into the U.S. national stage under 35 U.S.C. §371. The May 14, 1999 submission included a Transmittal Letter for a PCT International Application Entering the National Stage in the US as a Designated or Elected Office under 35 U.S.C. §371, a copy of the International Search Report, a copy of the International Preliminary Examination Report, and a check for \$1,200. A copy of the Transmittal Letter, referencing International Application No. PCT/GB97/03152, is attached hereto as Exhibit B.
- 4. On June 11, 1999, the U.S. Patent and Trademark Office mailed to the Applicants' representative a Notification of Missing Requirements under 35 U.S.C. §371, indicating that copies of International Application No. PCT/GB97/03152, the International Search Report, the International Preliminary Examination Report, and the priority document had been received from either Applicants or the International Bureau of the PCT. The Notification of Missing Requirements incorrectly referenced the international patent application as International Application No. PCT/GB97/03512. A copy of the Notification of Missing Requirements is attached hereto as Exhibit C.
- 5. On June 25, 1999, in response to the Notification of Missing Requirements, Applicants fulfilled the requirements of 35 U.S.C. §371 by submitting an executed Declaration (along with the required fee), which properly referenced the international patent application as International Application No. PCT/GB97/03152.
- 6. On August 11, 2000, the U.S. Patent and Trademark Office mailed to the Applicants' representative a Notification of Acceptance of Application for national patentability examination, which correctly referenced the international patent application as International Application No. PCT/GB97/03152. A copy of the Notification of Acceptance is attached hereto as Exhibit D.

Letter to Office of PCT Operations U.S. Serial No. 09/308,166 Atty Docket No. NNM-001 Page 2 of 3

- 7. On September 1, 2000, the Office of Initial Patent Examination issued a Filing Receipt for the national stage of the international patent application, which, in the Continuing Data as Claimed by Applicant section, correctly stated, "This Application is a 371 of PCT/GB97/03152 11/17/97" (emphasis added). Corrected versions of the Filing Receipt were issued per Applicants' requests on October 6, 2000, and February 26, 2001, each containing the same proper Continuing Data as Claimed by Applicant entry. A copy of the Corrected Filing Receipt dated February 26, 2001, is attached hereto as Exhibit E.
- 8. On December 4, 2000, Applicants submitted a Preliminary Amendment along with a Power of Attorney and Revocation of Prior Powers form, requesting that all future correspondence be addressed to:

Patent Administrator, Testa, Hurwitz & Thibeault, LLP High Street Tower 125 High Street Boston, MA 02110

- 9. On June 13, 2001, the Applicants' representative submitted a Status Inquiry form along with a Request to Correct Attorney of Record, resubmitting the Power of Attorney and Revocation of Prior Powers form initially filed on December 4, 2000, with the Preliminary Amendment. No response to the Status Inquiry has been received.
- 10. On June 14, 2002, following a conversation with a customer service representative at the PCT Help Desk, the undersigned attorney contacted Ms. Patricia Booker of the Office of PCT Operations who indicated that the national stage application, i.e. U.S. Serial No. 09/308,166, had not yet been forwarded for patentability examination and was marked for "troubleshooting," because there was a discrepancy in the PCT Serial Number. Specifically, Ms. Booker stated that the U.S. Serial No. 09/308,166, assigned to the above-identified international patent application following its entry into the national stage in the U.S., as indicated in the Filing Receipt, was allegedly associated with two international applications, International Application Nos. PCT/GB97/03512 and PCT/GB97/03152.
- 11. Ms. Booker requested that the undersigned attorney fax her a statement confirming the proper International Application Number of the international patent application. She indicated that upon receipt of such statement, she would rectify the discrepancy in the records of the U.S. Patent and Trademark Office and would promptly forward the above-identified national stage patent application to the Office of PCT Legal Administration for further processing.
- 12. Ms. Booker also indicated that neither the Power of Attorney and Revocation of Prior Powers form filed on December 4, 2000, nor the June 13, 2001 submission, was in the application file and requested that the undersigned attorney resubmit that submission so that the change in the attorney of record and the correspondence address could be processed. A copy of the June 13, 2001 submission is attached hereto as Exhibit F.
- 13. Per Ms. Booker's request, <u>Applicants confirm that the proper International Application Number for the international patent application in question is PCT/GB97/03152</u>. Applicants further state that the International Application No. PCT/GB97/03512 (now U.S. Patent No. 6,348,203, issued February 19, 2002, to Goodman *et al.*) was not filed by Applicants and is not related in any way to International Application No. PCT/GB97/03152.
- 14. Accordingly, in light of the substantial delay in processing of the above-identified national stage patent application, which occurred through no fault of Applicants, Applicants respectfully request expedited handling of this matter.

Letter to Office of PCT Operations U.S. Serial No. 09/308,166 Atty Docket No. NNM-001 Page 3 of 3

- 15. Applicants understand that this application is presently in good standing, is not abandoned, and in condition for examination.
- 16. Applicants note that U.S. Patent Application Serial No. 09/730,117, which is a continuation of the above-identified national stage patent application, already has been transferred to Group 1773 for examination and is assigned to Examiner Resan. To expedite prosecution of these related applications, Applicants respectfully request that the above-identified national stage patent application also be assigned to Examiner Resan in Group 1773.
- 17. Applicants believe that no fee is due upon filing of this submission. However, should any fee be required, the Director is authorized to charge Deposit Account No. 20-0531 for the required fee.

Respectfully submitted,

Mark L. Beloborodov, Reg. No. 50,773

Attorney for Applicants

Testa, Hurwitz, & Thibeault, LLP

High Street Tower 125 High Street

Boston, Massachusetts 02110

Date: June 19, 2002

Tel. No. (617) 248-7453 Fax: (617) 248-7100

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WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6: WO 98/22942 (11) International Publication Number: G11B 5/712, 5/62 (43) International Publication Date: 28 May 1998 (28.05.98) PCT/GB97/03152 (81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, (21) International Application Number: BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, 17 November 1997 (17.11.97) GH, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, (22) International Filing Date: LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, (30) Priority Data: 16 November 1996 (16.11.96) ✓ GB 9623851.4 BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, Eric, (71)(72) Applicants and Inventors: MAYES, -{US/GB}; 5 Brock Street, Bath BA1 26N (GB). TYLER, ML, MR, NE, SN, TD, TG). Malvin, Nicolas [GB/GB]; 9 The Circus, Bath BA1 2EW Published (74) Agent: NASH, David, Allan; Haseltine Lake & Co., Imperial With international search report. House, 15-19 Kingsway, London WC2B 6UD (GB).

(54) Title: MAGNETIZABLE DEVICE

(57) Abstract

Theres is disclosed a magnetic recording medium which includes a magnetizable layer thereon, wherein said magnetizable layer comprises a plurality of ferri- or ferromagnetic particles each having a largest dimension no greater than 100nm, and each of which particles represents a separate ferromagnetic domain.

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TRANSMITTAL LETTER FOR A PCT INTERNATIONAL APPLICATION
ENTERING THE NATIONAL STAGE IN THE U.S.
AS A DESIGNATED OF ELECTED OFFICE UNDER 35 USC 371

EXHIBIT B

Attorney's Docket No.: HASLP003

Date: May 14, 1999

Express Mail" mailing label number (from mail label): EL243914071

Express Mail No. EL956539515US

Date of Deposit: May 14, 1999

Information Disclosure Statement

I hereby certify that this paper is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service, as required under 37 CFR 1.10, on the date indicated above and is addressed to the Assistant Commissioner for Patents, Box PCT Application, Washington, D.C. 20231.

Name:	Dionna Holmes	BEYER & WEAVER, LLP					
Signature:	Droma Admos						
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Assistant Commissioner	for Patents	9 · 1 ·					
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Attention: DO/EO/US		<i>y</i> c. <i>y</i>					
	rewith are the papers required to the following PCT international pates	enter the national state in the U.S. as a designated tent application:					
INTERNATIONAL Int'l Filing Date:	L APPLICATION NUMBE 17 November 1997	R: PCT/GB97/03152					
1st Priority Date:	16 November 1996						
Inventor(s):	MAYES, Eric, Leigh						
_	TYLER, Malvin, Nicolas						
For:	MAGNETIZABLE DEVI	CE					
The United States Patent	t Office is: (select one)						
	Office (No Demand was filed - See 37	CFR 1.494)					
X An Elected Off	ice (A Demand for Preliminary Examin	nation was Filed - See 37 CFR 1.495)					
Enclosed are:							
than delay exam	This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).						
	A copy of the international application (if this line is not checked, the international application was previously communicated by the International Bureau or the international application was originally filed in the						
USPTO).	USPTO).						
An English Tra	An English Translation of the International Application						
A Combined D	A Combined Declaration and Power of Attorney A copy of amendments made under PCT Article 19						
A translation of	f amendments made under PCT Art						
	f annexes to the international prelim						
Verified Statem	nent establishing Small Entity Statu	•					
An Assignment	t of the Invention to: ***.						
(with S	\$40.00 recordal fee)						

A Preliminary Amendment A copy of the International Search Report A copy of the Preliminary Examination Report A check to cover the filing fees (including the basic national fee under 37 CFR 1.492(a)) in the amount calculated below: **FEE CALCULATION** <u>X</u> **BASIC FEE** \$930 (IPEA-U.S. \$720/360; ISA-U.S. \$790/395; PTO not ISA or IPEA \$1070/535; U.S. IPEA all claims meet 33(2)-(4) \$98/49; File w/ EPO or JPO search report \$930/465;) Surcharge for filing a late oath or declaration (\$130/65) Surcharge for filing a late translation (\$130) Multiple dependent claims (\$270/135) \$ 270 Excess claims - see calculation below \$ *** **Total Claims:** 16 -20 = 0 X \$22/11 claim \$ -0-Independent Claims: 2 -X \$82/41 ind. claim = \$ -0-**Excess Claim Total** \$ -0-Assignment recordal fee (\$40) TOTAL FEES \$1200

Please direct any correspondence to:

Customer Number: 022434
BEYER & WEAVER, LLP P.O. Box 61059 Palo Alto, CA 94306

PATENT TRADEHARK OFFICE

The Commissioner is hereby authorized to charge any additional fees or credit any overpayment to Deposit Account No. 50-0388. A duplicate copy of this transmittal is enclosed.

Respectfully submitted.

Jersim Vu

Joseph M. Villeneuve

Registration No. 37,460

BEYER & WEAVER, LLP P.O. BOX 61059 Palo Alto, CA 94306 (650) 493-2100

U.S. APPLICATION NO.	FIRST NAMED A	APPLICANT	ATTY, DOCKET NO.
09/308166	MAYES	E	HASLP003
BEYER & WEAVER LLP	1	INTERNAT	TIONAL APPLICATION NO.
P O BOX 61059		PC	T/GB97/03512
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Preliminary amendment(s) filed	and		
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Assignment document.	••		
Power of Attorney and/or Change of Act	idress.		
☐ Verified Statement Claiming Small Enti	ty Status		
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Copy of the International Search Report	and copies of the reference	s cited therein.	
└ Other:			••
The following items MUST be furnished wire acceptance under 35 U.S.C. 371:	thin the period set forth below	in order to comple	te the requirements for
a. Translation of the application into En	glish Note a processing fee w	ill be required if a	whenistad later than the
appropriate 20 or 30 months from the pr	riority date.	m oe required it s	donnited later than the
☐ The current translation is de		cated on the atta	ched Notice of Defective
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b. Processing fee for providing the trans 30 months from the priority date (37 CF	lation of the application and/or R 1.492(f)).	r the Annexes late	r than the appropriate 20 or
c. Oath or declaration of the inventors,	in compliance with 37 CFR 1.4	497(a) and (b), ide	ntifying the application by
the International application number and	international filing date.		
The current oath or declaration on the attached PCT/DO/EO/9	does not comply with 37 CFR	1.497(a) and (b)	for the reasons indicated
d. Surcharge for providing the oath or d		priate 20 or 30 mo	nths from the priority date
(37 CFR 1.492(e)).			
3. Additional claim fees of \$ as	a 🗆 large entity 🗆 small enti	ty, including any	required multiple dependent
claim fee, are required. Applicant must submit tue. See attached PTO-875.	the additional claim fees or car	ncel the additional	claims for which fees are
ALL OF THE ITEMS SET FORTH IN 2(a)-2	k(d) AND 3 ABOVE MUST B	E SUBMITTED	WITHIN ONE MONTH
FROM THE DATE OF THIS NOTICE OR E THE APPLICATION, WHICHEVER IS LAT	SY L. 21 OR LE 31 MONTHS	FROM THE PR	HORITY DATE FOR
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The time period set above may be extended by a CFR 1.136(a).	filing a petition and fee for exte	ension of time und	er the provisions of 37
CI R 1.150(a).			
4. Translation of the Annexes MUST be submit	tted no later that the time perio	d set above or the	annexes will be cancelled.
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5. The Article 19 amendments are cancelled 194(d)) or 30 (37 CFR 1.495(d)) months from the	since a translation was not pro	vided by the appro	opriate 20 (37 CFR.
Applicant is reminded that any communication to	o the United States Patent and	Trademark Office	must be mailed to the
ddress given in the heading and include the U.S			
A copy of this notice MUS:	T be returned with	this respon	se.
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UNITED STATES DEPARTMENT OF COMMERCE Patent and Trademark Office ddress: ASSISTANT COMMISSIONER FOR PATENTS

n9/308/66		Washington, D.C. 2	10231 B. F. (R.CA
U.P. DUPLICATION NO.	MAYES FRET HAME	APPLICANT ATTY	Y.BOCKET NO.
022434 BEYER WEAVER & THOMAS P O BOX 130 MOUNTAIN VIEW CA 94042		LA PENGDATE 11/17/5	PELORITY DATE

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	- DATE MARES: 08/14/00
NOTIFICATION OF ACCEPTANCE OF APPLICAT	FION UNDER 35 U.S.C. 371
AND 37 CFR 1.494 OR 1.49	95
1. The applicant is hereby advised that the United States Patent and Trac	demark Office in its capacity asa
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2. The United States Application Number assigned to the application is	shown shove and the relevant dates and
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3. A request for immediate examination under 35 U.S.C. 371(f) wa	
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Copy of the Annexes to the International Preliminary Examinat	ion Report (IPER).
Translation of Annexes to the IPER into English.	
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Information Disclosure Statement(s) filed	and
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Power of Attorney and/or Change of Address.	•
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Applicant is reminded that any communication to the United States Patent and Trademark Office must be mailed to the address given in the heading and include the U.S. application no shown above. (37 CFR 1.5)

FORM PCT/DO/EO/903 (December 1997)

Telephone: 703

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WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

i) International Patent Classification ⁶ :		(11) International Publication Number:	WO 98/22942
G11B 5/712, 5/62	A1	(43) International Publication Date:	28 May 1998 (28.05.98)
21) International Application Number: PCT/GE 22) International Filing Date: 17 November 1997 (20) 30) Priority Data: 9623851.4 16 November 1996 (16.11.5) 31)(72) Applicants and Inventors: MAYES, Eric [US/GB]; 5 Brock Street, Bath BA1 26N (GB). Malvin, Nicolas [GB/GB]; 9 The Circus, Bath E (GB). 34) Agent: NASH, David, Allan; Haseltine Lake & Co. House, 15–19 Kingsway, London WC2B 6UD (GB).	(17.11.9) (17.11.9) (17.11.9) (17.11.9) (17.11.9) (17.11.9) (17.11.9) (17.11.9)	BY, CA, CH, CN, CU, CZ, DE GH, HU, ID, IL, IS, JP, KE, K LR, LS, LT, LU, LV, MD, MG NZ, PL, PT, RO, RU, SD, SE, S TT, UA, UG, US, UZ, VN, YU KE, LS, MW, SD, SZ, UG, ZW, BY, KG, KZ, MD, RU, TJ, TM) CH, DE, DK, ES, FI, FR, GB, PT, SE), OAPI patent (BF, BJ, ML, MR, NE, SN, TD, TG). Published	, DK, EE, ES, FI, GB, GE, GG, KP, KR, KZ, LC, LK, MK, MN, MW, MX, NO, G, SI, SK, SL, TJ; TM, TR, ZW, ARIPO patent (GH), Eurasian patent (AM, AZ), European patent (AT, BE, GR, IE, IT, LU, MC, NL, CF, CG, CI, CM, GA, GN

(57) Abstract

Theres is disclosed a magnetic recording medium which includes a magnetizable layer thereon, wherein said magnetizable layer comprises a plurality of ferri- or ferromagnetic particles each having a largest dimension no greater than 100nm, and each of which particles represents a separate ferromagnetic domain.

MAGNETIZABLE DEVICE

This invention relates to a magnetizable device which comprises a magnetic layer composed of domain-separated, nanoscale (e.g. 1-100nm) ferromagnetic particles. The magnetizable device of the invention may be used as a magnetic storage device having improved data storage characteristics. In particular, the invention relates to magnetic storage media comprising single-domain, domain-separated, uniform, ferromagnetic nanoscale (e.g. 1-100 nm) particles which may be arranged into a regular 2-D packed array useful in the storage of information.

Among the possible pathways to ultrahigh-density (>=1 Gbit/in²) magnetic media is the use of nanoscale 15 (1-100 nm) particles. Beyond the standard requirements for magnetic media, a viable particulate media should have a small standard deviation in particle size as well as the particles being exchange decoupled. requirements are necessary to avoid adverse media 20 noise. Current methods of fabricating nanoscale particles, such as arc-discharge or multiple target ion-beam sputtering, have not fully addressed these two Moreover, if the uniform particles are requirements. arranged into an ordered array, each particle can represent a "bit" of information at a predictable location further increasing the media's efficiency. This invention details methods of producing particulate media that meet these requirements for ultrahighdensity recording. This invention is also an open system which allows for the production of a variety of magnetic materials, such that the media can be tuned for different applications.

In particular this invention details the use of an iron storage protein, ferritin, whose internal cavity is used to produce the nanoscale particles. Ferritin is utilised in iron metabolism throughout living

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species and its structure is highly conserved among It consists of 24 subunits which are arranged to provide a hollow shell roughly 8 nm in diameter. The cavity normally stores 4500 iron(III) atoms in the form 5 of paramagnetic ferrihydrite. However, this ferrihydrite can be removed (a ferritin devoid of ferrihydrite is termed "apoferritin") and other materials may be incorporated. Examples include ceramics, superparamagnetic magnetite, acetaminophen, 10 and even the sweetener aspartame. To address magnetic media concerns, the invention incorporates ferromagnetically ordered materials.

According to a first aspect of the present invention, there is provided a magnetizable device 15 which comprises a magnetic layer composed of domainseparated, ferromagnetic particles each of which has a largest dimension no greater than 100nm.

According to a second aspect of the invention, there is provided a magnetic recording medium which includes a magnetizable layer, wherein said magnetizable layer comprises a plurality of ferromagnetic particles each having a largest dimension no greater than 100nm, and each of which particles represents a separate ferromagnetic domain. magnetizable layer is preferably supported on a non-25 magnetic substrate.

According to a third aspect of the present invention, there is provided a magnetic composition comprising a plurality of ferromagnetic particles each 30 of which is bound to an organic macromolecule, and each of which has a largest dimension no greater than 100nm. In this aspect of the invention, it is preferred that said organic macromolecule is ferritin from which the normal core ferrihydrite has been removed and replaced 35 by a ferromagnetic particle.

As used herein, the term "ferromagnetic" embraces

materials which are either "ferromagnetic" and "ferrimagnetic". Such usage is common in the electrical engineering art.

The ferromagnetic particles used in the invention should be of a material and size such that they possess ferromagnetic properties at ambient temperatures (e.g. 15°C to 30°C),

Preferably, the ferromagnetic particles each have a largest dimension no greater than 50nm, more preferably less than 25nm and most preferably smaller than 15nm. The largest dimension of the ferromagnetic particles should not be so small that the particle will lose its ferromagnetic property and become superparamagnetic at the desired operating temperature of the recording medium. Typically, for operation at ambient temperature, this means that the magnetic particles will normally be no smaller than about 3nm in their largest diameter.

In the magnetizable device of the first aspect of this invention and the magnetic recording medium of the 20 second aspect of this invention, the distance between adjacent ferromagnetic domains is preferably as small as possible to permit the maximum number of discrete domains in a given area, and provide the maximum storage capacity for the recording medium. The actual lower limit will vary for different materials and other conditions such as the temperature at which the recording medium is to be used. The key requirement, however, is that neighbouring domains should not be able to interfere magnetically with each other to the extent that the magnetic alignment of any domain can be altered by neighbouring domains. Typically, the lower limit on the spacing of the domains is about 2nm. distance between adjacent domains will be determined by 35 the density of discrete domains required. Typically, however, to take advantage of the miniaturization

possibilities provided by the invention, the distance between adjacent domains will be no greater than 10nm.

Generally the particles will be uniform in size, by which we mean that the particles do not vary in largest diameter by more than about 5%. One of the advantages of the use in the invention of an organic macromolecule which binds a magnetic particle by surrounding it is that this can be used to select particles of a uniform size.

In the case where the particles are spheroidal, it will be the diameter of the particles which must be no greater than 100nm.

In preferred embodiments of all aspects of this invention, each ferromagnetic particle is encased, or partially encased, within an organic macromolecule. The term macromolecule means a molecule, or assembly of molecules, and may have a molecular weight of up 1500kD, typically less than 500kD. Ferritin has a molecular weight of 400kD.

The macromolecule should be capable of binding by encasing or otherwise organising the magnetic particle, and may therefore comprise a suitable cavity capable of containing the particle; a cavity will normally be fully enclosed within the macromolecule.

25 Alternatively, the macromolecule may include a suitable opening which is not fully surrounded, but which nevertheless is capable of receiving and supporting the magnetic particle; for example, the opening may be that defined by an annulus in the macromolecule. For

example, suitable macromolecules which may be used in the invention are proteins, for example the protein apoferritin (which is ferritin in which the cavity is empty), flagellar L-P rings, cyclodextrins, selfassembled cyclic peptides. As an alternative to

35 encasing the magnetic particles within the macromolecule, they may be organised on the

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macromolecule, such as on a bacterial S-layer.

Other materials which may be used in the invention to organise the ferromagnetic particles are inorganic-silica networks such as MCM type materials, dendrimers and micellar type systems.

The presently preferred macromolecule for use in the invention is the apoferritin protein which has a cavity of the order of 8nm in diameter. The ferri- or ferromagnetic particles to be accommodated within this protein should have a diameter no greater than 8nm.

The bound particles of this aspect of the present invention with a coating that inhibits aggregation and oxidation, also helping them to be domain-separated.

In the magnetizable device of the first aspect of this invention and the magnetic recording medium of the second aspect of this invention, the particles are preferably arranged in a 2-D ordered array which would yield an ultrahigh-density magnetic media.

The ferromagnetic material may be a metal, such as cobalt, iron, or nickel; a metal alloy, such as an alloy which contains aluminium, barium, bismuth, cerium, chromium, cobalt, copper, iron, manganese, molybdenum, neodymium, nickel, niobium, platinum, praseodymium, samarium, strontium, titanium, vanadium, ytterbium, yttrium or a mixture thereof; a metal ferrite such as a ferrite containing barium, cobalt, or strontium; or an organic ferromagnetic material.

When generating nanoscale particles, one major concern is that the particles produced are not superparamagnetic. Superparamagnetic particles are those which have permanent magnetic dipole moments, but the moments' orientations with respect to the crystallographic axes fluctuate with time. This is not useful for a practical magnetic storage media.

5 Superparamagnetism depends on the volume, temperature, and anisotropy of the particles. Via energy

considerations, one can derive an equation relating these quantities. The volume at which a particle becomes superparamagnetic (V_p) is given by: $V_p =$ 25kT/K, where k is Boltzman's constant, T the 5 temperature of the particle in degrees Kelvin, and K the anisotropy constant of the material. Using this formula, it is possible to determine the temperature at which a particle becomes superparamagnetic (the "blocking temperature") for a given material at a fixed 10 volume. In our specific case, the fixed volume is 8 nm in ferritin. If a cobalt metal particle with only crystalline anisotropy (that value being 45×10^5) is a sphere with a diameter of 8 nm, the blocking temperature is 353 °K. This is within the range of temperatures experienced within a hard disk drive, and the cobalt particles may prove to be a useful storage medium. Obviously, there are other considerations such as the materials' coercivity, moment, saturation magnetisation, and relaxation time. By tuning the materials incorporated into the ferritin, though, these 20 can be addressed.

Ferritin is utilised in iron metabolism throughout living species and its structure is highly conserved among them. It consists of 24 subunits arranged in a 432 symmetry which provide a hollow shell roughly 8 nm in diameter. The cavity normally stores 4500 iron(III) atoms in the form of paramagnetic ferrihydrite. However, this ferrihydrite can be removed (a ferritin devoid of ferrihydrite is termed "apoferritin") and other materials may be incorporated. The subunits in ferritin pack tightly, however there are channels into the cavity at the 3-fold and 4-fold axes. Lining the 3-fold channels are residues which bind metals such as cadmium, zinc, and calcium. By introducing such divalent ions one can potentially bind ferritin molecules together, or at least encourage their

35

proximal arrangement.

One method of preparing a 2-D packed array of ferromagnetically ordered particles of uniform size up to 8 nm includes the removal of the ferrihydrite core from the native ferritin in aqueous solution, the incorporation of ferromagnetically ordered cobalt metal particles by sodium borohydride reduction of the aqueous Co(II) solution into the ferritin cavities, the generation of a narrow size distribution through 10 ultracentrifugation, the injection of particles into an MES/glucose subphase solution upon which the 2-D array assembles, and the transfer of the 2-D array to a substrate which is then carbon coated. In this method, the ferritin source may be a vertebrate, invertebrate, plant, fungi, yeast, bacteria, or one produced through recombinant techniques.

In the method described, a metal alloy core may be produced by sodium borohydride reduction of a water soluble metal salt. Other oxidation methods include carbon, carbon monoxide, hydrogen, or hydrazine hydrate solution. Alternatively, a suitable solution may be oxidised to yield a metal ferrite core. Oxidation may be chemical or electrochemical to yield the metal ferrite.

In this method, other methods of selecting a narrow size distribution may be employed such as short or long column meniscus depletion methods or magnetic field separation.

Further, in this method, divalent metal salts

30 containing cadmium, calcium, or zinc may be added into
the subphase solution to aid in particle ordering.

Further, in this, other methods of arranging the particles into a 2-D array may be employed, such as solution evaporation onto a solid substrate.

Further, in this method, the 2-D array may be coated with carbon-based films such as hydrogenated or

nitrogen doped diamond-like carbon, or with siliconbased films such as silicon dioxide.

In the present invention, ferritin may be used to enclose a ferromagnetic particle whose largest 5 dimension is limited by ferritin's inner diameter of 8 The particles are produced first by removing the ferrihydrite core to yield apoferritin. The is done by dialysis against a buffered sodium acetate solution under a nitrogen flow. Reductive chelation using 10 thioglycolic acid is used to remove the ferrihydrite core. This is followed by repeated dialysis against a sodium chloride solution to completely remove the reduced ferrihydrite core from solution. Once the apoferritin is produced, ferri- or ferromagnetic particles are incorporated in the following ways. first is by reducing a metal salt solution in the presence of apoferritin. This is performed in an inert atmosphere to protect the metal particles from oxidation which would lessen their magnetic benefit. combination of metal salts in solution can also be 20 reduced to generate alloys or alloy precursors. Sintering or annealing in a magnetic field may be necessary to generate the useful magnetic alloys. Another method is to oxidise a combination of an 25 iron(II) salt and another metal salt. This gives a metal ferrite particle which does not suffer negatively from oxidation. The metal salts which are beneficial include salts of aluminium, barium, bismuth, cerium, chromium, cobalt, copper, iron, manganese, molybdenum, 30 neodymium, nickel, niobium, platinum, praseodymium, samarium, strontium, titanium, vanadium, ytterbium, and yttrium.

A narrow size distribution of particles is necessary to avoid media noise. Such a distribution 35 can be obtained through a variety of procedures including, but not limited to, density gradient centrifugation or magnetic field separation.

While the production procedure detailed uses native horse spleen ferritin, this invention should not be seen as limited to that source. Ferritin can be found in vertebrates, invertebrates, plants, fungi, yeasts, bacteria, or even produced through recombinant techniques. By creating mutant apoferritins lacking the divalent binding site, others have found that the mutant proteins assemble into oblique assemblies as opposed to the regular hexagonal close-packed.

While ferritin seems to be an ideal system for generating nanoscale particles, it is not the only system available. For example, flagellar L-P rings are tubular proteins with an inner diameter of 13 nm. By creating a 2-D array of these proteins, metal films could be deposited into the tubular centres to create perpendicular rods of magnetic material. Also metal reduction in the presence of a microemulsion can be used to generate nanoscale particles which are coated with surfactant. This invention is open to other nanoscale particle production methods.

Finally an ordered arrangement of the particles is desired. One way to accomplish this is by injecting an aqueous solution of particles into an MES/glucose

25 subphase solution contained in a Teflon trough. The particles spread at the air-subphase interface, and a portion denature to form a monolayer film. The 2-D arrangement of encased particles occurs underneath this monolayer. After 10 minutes at room temperature, the arrangement and monolayer are transferred to a substrate by placing the substrate directly onto the monolayer for 5 minutes. After withdrawing the substrate, the attached arrangement is coated with a thin layer of carbon for protection. Other methods

35 such as solution evaporation onto a solid substrate can also give 2-D arrangements, and this invention should

not be seen as limited in its arrangement methods.

EXAMPLE 1

This example illustrates the preparation of apoferritin from horse spleen ferritin. Apoferritin

5 was prepared from cadmium-free native horse spleen ferritin (CalBiochem, 100 mg/ml) by dialysis (molecular weight cut-off of 10-14 kDaltons) against sodium acetate solution (0.2 M) buffered at pH 5.5 under a nitrogen flow with reductive chelation using

10 thioglycolic acid (0.3 M) to remove the ferrihydrite core. This is followed by repeated dialysis against sodium chloride solution (0.15 M) to completely remove the reduced ferrihydrite core from solution.

EXAMPLE 2

This example illustrates the preparation of cobalt metal within apoferritin. The apoprotein is added to a deaerated TES/sodium chloride solution (0.1/0.4 M) buffered at pH 7.5 to give an approximate 1 mg/ml working solution of the protein. A deaerated cobalt(II) [for example, as the acetate salt] solution (1 mg/ml) was added incrementally such that the total number of atoms added was approximately 500 atoms/apoprotein molecule. This was allowed to stir at room temperature for one day in an inert atmosphere.

This is followed by reduction of the cobalt(II) salt with sodium borohydride to cobalt(0) metal. The final product yielded a solution of cobalt particles, each surrounded by a ferritin shell.

EXAMPLE 3

This example illustrates the preparation of a metal alloy such as yttrium cobalt (YCo₅) within apoferritin. The metal alloy follows the same procedure as Example 2 but using a 1:5 ratio of yttrium(III) [for example, as the acetate salt] to cobalt(II) [for example, as the acetate salt]. The final product yielded a solution of yttrium cobalt particles, each

surrounded by a ferritin shell.

EXAMPLE 4

This example illustrates the preparation of a metal ferrite such as cobalt ferrite (CoO·Fe₂O₃) within apoferritin. The apoprotein is added to a deaerated MES/sodium chloride solution (0.1/0.4 M) buffered at pH 6 to give an approximate 1 mg/ml working solution of the protein. A deaerated solution of cobalt(II) [for example, as the acetate salt] and iron(II) [for 10 example, as the ammonium sulphate salt] in a ratio of 1:2 is added incrementally and allowed to air-oxidise. The final product yielded a solution of cobalt ferrite particles, each surrounded by a ferritin shell.

EXAMPLE 5

This example illustrates the 2-D arrangement of 15 ferritin-encased magnetic particles. An aqueous solution of particles [from Examples 2-4, and whose uniformity in size has been selected] is injected into an MES/glucose subphase solution (0.01 M/2%) contained in a Teflon trough. The particles spread at the air-20 subphase interface, and a portion denature to form a monolayer film. The 2-D arrangement of encased particles occurs underneath this monolayer. After 10 minutes at room temperature, the arrangement and monolayer are transferred to a substrate by placing the substrate directly onto the monolayer for 5 minutes. After withdrawing the substrate, the attached arrangement is coated with a thin layer of carbon for protection.

CLAIMS:

- A magnetizable device which comprises a
 magnetic layer composed of domain-separated,
 ferromagnetic particles each of which has a largest
 dimension no greater than 100nm.
 - 2. Magnetic recording medium which includes a magnetizable layer thereon, wherein said magnetizable layer comprises a plurality of ferromagnetic particles each having a largest dimension no greater than 100nm, and each of which particles represents a separate ferromagnetic domain.
 - 3. Magnetic recording medium according to claim 2, wherein the distance between adjacent ferromagnetic domains is at least 2nm.
- 4. Magnetic recording medium according to claim
 2 or 3, wherein the distance between adjacent
 ferromagnetic domains is no greater than 10nm.
- 5. Magnetic recording medium according to claim 1, 2, 3 or 4, wherein each ferromagnetic particle is 20 encased within an organic macromolecule.
 - 6. Magnetic recording medium according to claim 5, wherein each ferromagnetic particle is encased within the cavity or opening of a protein macromolecule.
- 7. Magnetic recording medium according to claim 6, wherein each ferri- or ferromagnetic particle is encased within an apoferritin protein.
- 8. A magnetic composition comprising a plurality of ferromagnetic particles each of which is bound to an organic macromolecule, and each of which ferromagnetic particles has a largest dimension no greater than 100nm.



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APPLICATION NUMBER	FILING DATE	GRP ART UNIT	FIL FEE REC'D	ATTY.DOCKET.NO	DRAWINGS	TOT CLAIMS	IND CLAIMS
09/308,166	06/25/1999	2754	615	HASLP003		32	2 .

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CORRECTED FILING RECEIPT

OC000000005797587

Date Mailed: 02/26/2001

Receipt is acknowledged of this nonprovisional Patent Application. It will be considered in its order and you will be notified as to the results of the examination. Be sure to provide the U.S. APPLICATION NUMBER, FILING DATE, NAME OF APPLICANT, and TITLE OF INVENTION when inquiring about this application. Fees transmitted by check or draft are subject to collection. Please verify the accuracy of the data presented on this receipt. If an error is noted on this Filing Receipt, please write to the Office of Initial Patent Examination's Customer Service Center. Please provide a copy of this Filing Receipt with the changes noted thereon. If you received a "Notice to File Missing Parts" for this application, please submit any corrections to this Filing Receipt with your reply to the Notice. When the PTO processes the reply to the Notice, the PTO will generate another Filing Receipt incorporating the requested corrections (if appropriate).

Applicant(s)

Eric Leigh Mayes, Bath, GBN; Malvin Nicolas Tyler, Bath, GBN;

Continuing Data as Claimed by Applicant

THIS APPLICATION IS A 371 OF PCT/GB97/03152 11/17/1997

Foreign Applications

UNITED KINGDOM 9623851.4 11/16/1996

If Required, Foreign Filing License Granted 09/01/2000

Projected Publication Date:

Non-Publication Request: No

No Docketing Necessary

Early Publication Request: No

Date Date

Title

Magnetizable Device

Reviewed & Approved

Resp. Atty

Date

Preliminary Class

360

Data entry by : SHEFFEY, CATHERINE

Team : OIPE

Date: 02/26/2001

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NNM-001 (7596/1)

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Certificate of First Class Mailing Under 37 C.F.R. 1.8 (1 pg.); Transmittal (1 pg.); Status Inquiry and Request to Correct Attorney of Record (2 pgs.); Copy of Power of Attorney by Assignee of Entire Interest, Revocation of Prior Powers and New Power of Attorney (3 pgs.); Copy of Return Receipt Postcard Date-Stamped 12/4/00 (1 pg.) and Return Receipt Postcard.

Name of Applicants: Mayes and Tyler

Intf. or Serial Number: 09/308,166

Atty: TATurano/MLBeloborodov

Date: June 13, 2001 BELOBOM\7596\1.2115381_1



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANT(S):

Mayes and Tyler

SERIAL NO.:

09/308,166

GROUP NO.:

2754

FILING DATE:

June 25, 1999

EXAMINER:

Not Yet Assigned

TITLE:

MAGNETIZABLE DEVICE

CERTIFICATE OF FIRST CLASS MAILING UNDER 37 C.F.R. 1.8

I hereby certify that this correspondence, and any document(s) referred to as enclosed herein, is/are being deposited with the United States Postal Service as first class mail, postage prepaid, in an envelope addressed to the Assistant Commissioner for Patents, Washington, DC 20231 on this 13th day of June, 2001.

Assistant Commissioner for Patents Washington, D.C. 20231

Sir:

Submitted herewith are: Transmittal (1 pg.); Status Inquiry and Request to Correct Attorney of Record (2 pgs.); Copy of Power of Attorney by Assignee of Entire Interest, Revocation of Prior Powers and New Power of Attorney (3 pgs.); Copy of Return Receipt Postcard date-stamped 12/4/00 (1 pg.) and Return Receipt Postcard.

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			Patent No.		Not ap	plicable	
			Issue Date		Not ap	plicable	
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	Fee Transmittal Form		Parts of Application	on (PTO-1553)	_	of Patent Appeals and Interferences	
	☐ Check Attached☐ Copy of Fee Transmittal Form		Formal Drawing(s)		Appeal Brief (in triplicate)	
□] Amendment/Response		Request For Cont Examination (RC			Status Inquiry	
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	Affidavits/declaration(s) Letter to Official Draftsperson		Power of Attorney (Revocation of Prior Powers)		×	Certificate of First Class Mailing under 37 C.F.R. 1.8	
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1	Information Disclosure		Small Entity Sta	tement			١
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ŀ	CORRESPONDENCE ADDRESS			SIGNATURE B	LOCK	Respectfully submitted,	
	Direct all correspondence to: Patent Administra Testa, Hurwitz & High Street Towe 125 High Street Boston, MA 021 Tel. No.: (617) 2 Fax No.: (617) 2		Date: June 13, 20 Reg. No. 35,722 Tel. No.: (617) 24 48-7000 Take: June 13, 20 Reg. No. 35,722 Tel. No.: (617) 24		48-7738	Thomas A Turano Attorney for Applicants Testa, Hurwitz & Thibeault, LLP High Street Tower 125 High Street Boston, MA 02110	

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANT(S):

Mayes and Tyler

SERIAL NO.:

09/308,166

GROUP NO.:

2754

FILING DATE:

June 25, 1999

EXAMINER:

Not Yet Assigned

TITLE:

MAGNETIZABLE DEVICE

Assistant Commissioner for Patents Washington, D.C. 20231

STATUS INQUIRY AND REQUEST TO CORRECT ATTORNEY OF RECORD

- 1. Almost two years passed since the filing of the above-referenced application on June 25, 1999. No communication has been received from the Patent and Trademark Office indicating action on this application to date.
- On June 12, 2001, a representative of the undersigned contacted the Customer Service Desk for the Technology Center 2700, and was advised by Mr. Tim Vo that, according to the records of the U.S. Patent and Trademark Office ("USPTO"), the attorneys of record for the above-referenced application were Beyer & Weaver, LLP of Mountain View, CA.
- 3. On November 15, 2000, Applicants revoked the powers of attorney previously given, and appointed the attorneys and agents of the law firm of Testa, Hurwitz & Thibeault, LLP to prosecute the above-referenced application. Revocation of Prior Powers and New Power of Attorney form was received by the USPTO on December 4, 2000. A copy of the form and the stamped return receipt postcard are attached hereto.
- 4. Kindly update the records of the USPTO accordingly, and advise the undersigned of the present status of this application. A stamped return-addressed envelope is provided.

Status Inquiry and Request to Correct Attorney of Record Serial No. 09/308,166 Page 2 of 2

Respectfully submitted,

Date: June 13, 2001 Reg. No. 35,722

Tel. No.: (617) 248-7738 Fax No.: (617) 248-7100 Thomas A. Turano Attorney for Applicants

Testa, Hurwitz, & Thibeault, LLP

High Street Tower 125 High Street

Boston, Massachusetts 02110

BELOBOM\7596\1.2114646

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANT(S):

Mayes and Tyler

SERIAL NO.:

09/308.166

GROUP NO.:2754

FILED:

June 25,1999

EXAMINER: not yet assigned

TITLE:

MAGNETIZABLE DEVICE

Assistant Commissioner for Patents Washington, D.C. 20231

POWER OF ATTORNEY BY ASSIGNEE OF ENTIRE INTEREST REVOCATION OF PRIOR POWERS AND NEW POWER OF ATTORNEY

Sir:

As assignee of record of the entire interest of the above-identified

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patent,

all powers of attorney previously given are hereby revoked and

the following attorneys and/or agents are hereby appointed to prosecute and transact all business in the Patent and Trademark Office connected therewith.

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Power Of Attorney By Assignee Of Enti. Interest Revocation Of Prior Powers and New Power of Attorney Serial No. 09/308,166 Page 2 of 3

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Attached as part of this power of attorney is the authorization of the above-named attorneys/agents to accept and follow instructions from my representatives.

Assignee also hereby grants additional Powers of Attorney to the attorneys and/or agents named above to file and prosecute foreign national patent applications in any and all countries of the world, a regional patent application under the European Patent Convention and/or an international application under the Patent Cooperation Treaty based upon the above-identified application, including a power to meet all designated office requirements for designated states.

All future correspondence should be sent to:

Patent Administrator Testa, Hurwitz & Thibeault, LLP High Street Tower 125 High Street Boston, MA 02110 Power Of Attorney By Assignee Of Entire ' rest Revocation Of Prior Powers and New Power. of Attorney Serial No. 09/308,166 Page 3 of 3 The assignee of record of the entire interest of the above-identified application 図 patent is Name of assignee of entire interest Nanomagnetics Limited Recorded in PTO on Reel No.: Frame No.: Recorded herewith Respectfully submitted, Dated: Nov 15th Eric L. Mayes Technical Director Nanomagnetics Limited 9 The Circus, Bath BA1 2EW, Great Britain

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